#### DATA EVALUATION RECORD

Chemical Code 129103

STUDY 4

CHEM "Ag-Zn Zeolite"

Kyranos, J.N. 1991. Release of Silver and Zinc under Hydrolysis Conditions. Performed by Arthur D. Little, Cambridge, MA; ADL #66365-10; Completed 5/31/91. Submitted by Kanebo Zeolite USA, Inc., New York, NY.

Avg ust 18, 1993

MRID #42032806

REVIEWED BY: S.C. Termes, Chemist Review Section #3

OPP/EFED/EFGWB (703) 305-5243

## CONCLUSIONS

This study provides only ANCILIARY information at this time. There was apparent contamination of samples by zinc.

As indicated in the review of the "Ag-Cu Zeolite" (Study 1; MRID #41613816), the registrant must clarify which type of zeolite is used for exhange by silver and zinc. In addition, the oxidation state of silver in the parent "Ag-Zn Zeolite" must be clarified.

The registrant did not present the predominance of Ag and Zn species in the buffer systems used in the study. The data must be presented for both the the spiked solutions and the test solutions (refer to comments in Study 1).

For registration of potential OUTDOOR uses, refer to the "CONCLUSIONS" section in Study 1.

### Scientific Conclusions

The data presented in this study are of uncertain value (apparent contamination of samples by zinc). As with the "Ag-Cu Zeolite", the release of Ag and Zn appears to decrease with increasing pH. However, it appears that less Ag is released from "Ag-Zn Zeolite" than from the "Ag-Cu Zeolite".

#### MATERIALS AND METHODS

Test Material: A "Ag-Zn Zeolite" provided by registrant (zeolite type not specified) containing 3.6% Ag and 6.1% Zn by weight (anhydrous). The moisture content measured was measured by the performing laboratory as 16.8%.

<u>Test System</u>: The test sytem(s) consisted of 40-mL amber glass vials fitted with screww caps and Teflon-faced septa.

Each vial contained 30-50 mg sample of the "Ag-Zn Zeolite" and 30 mL of the desired buffer system (pH 5= acetate buffer; pH 7=

phosphate buffer; pH 9= borate buffer). The ratio of zeolite to buffer was about 1 mL/mg.

Vials were placed in a temperature controlled chamber (25±1 C) and protected from light. Samples were agitated with a magnetic stirrer.

The initial pH of the hydrolysis medium was measured with pH paper.

Control Samples: Negative (neat buffer solutions) and positive (buffer with Ag<sup>+</sup> or Zn<sup>2+</sup> nominal concentrations of 100 ppb and 50 ppb, respectively) were used. Initial spiked solutions contained both Ag and Zn, but the standard zinc solution (which was in 2% HCl) seemed to have affected the "recovery" of silver.

Sampling Times: 0, 2, 4, 8, 16, 24, and 30 days.

Analytical Methodology: After removal from vial, hydrolysates were passed through 0.45 ug Teflon filters to ensure that only dissolved silver and copper were measured; filtrates were stabilized with nitric acid and stored at 4 C prior to analysis.

Hydrolysates and negative and positive standards were analyzed by Inductively Coupled Argon Plasma (ICAP). Calibration curves and calculated sample concentrations were based on the average area of the ICP peak signal for three sequential aspirations.

All release data ws corrected to unit weight of zeolite powder,

ug/mL= ppb(ug/L) x 0.030L (hydrolysate volume)
Weight of zeolite present

# REPORTED RESULTS:

The release rates of Ag and Zn and % release at 30 days can be summarized as follows,

	Hq	5	<u>рн 7</u>		<u>pH 9</u>	
	Ag	<u>Zn</u>	Ag	<u>Zn</u>	Ag	<u>Zn</u>
Release Rate (ug/mg zeolite/day)	0.029	0.082	0.19	0.29	0.0047	0.030
% of Total Metal after 30-Days	2.4%	4.0%	0.53%	0.48%	0.39%	1.5%

The study was conducted at 25± 1 C for a 30-day period.

REVIEWER'S COMMENTS:

- 1. There was no attempt to present the predominance of Ag and Zn species in hydrosylates and "spiked" controls. This would have been useful in for knowing a priori which Ag or Zn species would be expected at the different pHs. This information is needed.
- 2. There was apparent contamination of samples by zinc.
- 3. The data from this study applies only to the type of zeolite used for exchange with Ag and Zn. The data may not apply for "Ag-Zn Zeolites" in which the zeolite is different.
- 4. The use of pH papers to measure pH is not a desirable method.

Table 1 - Sample Weights and pH Values for pH 5 Hydrolysis Samples

SAMPLE	POWDER WT (mg)*	INITIAL pH	TEST	TEST DATE	FINAL pH
HL-0A-5	35.49	5.0	0	3/25/91	5.0
HL-0B-5	32.05	5.0	0	3/25/91	5.0
HL-2A-5	38.05	5.0	2	3/27/91	5.0
HL-2B-5	29.86	5.0	2	3/27/91	5.0
HL-4A-5	29.10	5.0	4	3/29/91	5.0
HL-4B-5	30.06	5.0	4	3/29/91	5.0
HL-8A-5	35.70	5.0	8	4/2/91	5.6
HL-8B-5	30.73	5.0	8	4/2/91	5.6
HL-16A-5	42.29	5.0	16	4/10/91	5.0
HL-16B-5	29.97	5.0	16	4/10/91	5.0
HL-24A-5	27.82	5.0	24	4/18/91	5.0
HL-24B-5	24.36	5.0	24	4/18/91	5.0
HL-30A-5	32.97	5.0	30	4/24/91	5.0
HL-30B-5	28.21	5.0	30	4/24/91	5.0

<sup>\* -</sup> Anhydrous basis (corrected for 16.82% moisture)

Table 2 - Hydrolysate Silver and Zinc Concentrations for pH 5 Hydrolysis

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ZINC

					SILVER				ZINC		
SAMPLE	TEST	ANALYSIS DATE	DILUTION	SOL'N CONC (ppb)	AMNT RELSED (ug/mg)	AVERAGE (ug/mg)	RPD/RSD	SOL'N CONC (ppb)	AMNT RELSED (ug/mg)	AVERAGE (ug/mg)	RPD/RSI
HL-0A-5	0	3/29/91		594	0.50			592	0.50		
HL-0B-5	0	3/29/91		633	0.59	0.55	16	632	0.59	0.55	16
HL-2A-5	2	3/29/91		703	0.55			821	0.82		
						0.72	46			1.14	55
HL-2B-5	2	3/29/91		871	0.88			1444	1.45		
HL-4A-5	4	3/29/91		1321	1.36			1735	1.79		
HL-4A-5	4	3/29/91	1:5	1280	1.32			1740	1.79		
						1.33	4			1.77	2
HL-4B-5	4	3/29/91		1353	1.35			1775	1.77		
HL-4B-5	4	3/29/91	1:5	1275	1.27			1740	1.74		
HL-8A-5	8	4/9/91		931	0.78			1334	1.12		
HL-8A-5	8	4/9/91	1:10	1150	0.97			1310	1.10		
						0.97	13			1.39	24
HL-8B-5	8	4/9/91		1069	1.04			1747	1.71		
HL-8B-5	8	4/9/91	1:10	1100	1.07			1683	1.64		

<sup>\* -</sup> Outlier not used in calculating average

Table 2 - Hydrolysate Silver and Zinc Concentrations for pH 5 Hydrolysis (Continued)

SILVER	ZINC
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	TEST	ANALYSIS		SOL'N CONC	AMNT RELSED	AVERAGE	RPD/RSD	SOL'N CONC	AMNT RELSED	AVERAGE	RPD/RSD
SAMPLE	DAY	DATE	DILUTION	(ppb)	(ug/mg)	(ug/mg)		(ppb)	(ug/mg)	(ug/mg)	
HL-16A-5	16	4/15/91		496	0.35*			1144	0.81*		
HL-16A-5	16	4/15/91	1:10	626	0.44*			1031	0.73*		
HL-16B-5	16	4/15/91		1044	1.05	1.09	8	2428	2.43	2.34	8
HL-16B-5	16	4/15/91	1:10	1126	1.13			2235	2.24		
HL-24A-5	24	4/29/91		1687	1.82			4131	4.45		
HL-24A-5	24	4/29/91	1:10	1807	1,95	0.00		4055	4.37		
HL-24B-5	24	4/29/91		1570	1.93	2.00	10	3410	4.20	4.38	3
HL-24B-5	24	4/29/91	1:10	1854	2.28			3638	4.48		
HL-30A-5	30	4/29/91		1093	0.99			2499	2.27		
HL-30A-5	30	4/29/91	1:10	1283	1.17			2490	2.27		
HL-30B-5	30	4/29/91		1711	1.82	1.48	32	3775	4.01	3.12	57
HL-30B-5	30	4/29/91	1:10	1831	1.95			3679	3.91		

<sup>\* -</sup> Outlier not used in calculating average

Table 3 - Sample Weights and pH Values for pH 7 Hydrolysis Samples

SAMPLE	POWDER WT (mg)*	INITIAL pH	TEST	TEST DATE	FINAL pH
HL-0A-7	27.56	6.9	0	3/25/91	6.9
HL-0B-7	31.72	6.9	0	3/25/91	6.9
HL-2A-7	31.03	6.9	2	3/27/91	6.9
HL-2B-7	29.09	6.9	2	3/27/91	6.9
HL-4A-7	27.12	6.9	4	3/29/91	6.9
HL-4B-7	43.70	6.9	4	3/29/91	6.9
HL-8A-7	32.12	6.9	8	4/2/91	6.9
HL-8B-7	34.20	6.9	8	4/2/91	6.9
HL-16A-7	35.93	6.9	16	4/10/91	6.9
HL-16B-7	38.40	6.9	16	4/10/91	6.9
HL-24A-7	26.30	6.9	24	4/18/91	6.9
HL-24B-7	30.87	6.9	24	4/18/91	6.9
HL-30A-7	25.17	6.9	30	4/24/91	6.9
HL-30B-7	31.41	6.9	30	4/24/91	6.9

<sup>\* -</sup> Anhydrous basis (corrected for 16.82% moisture)

Table 4 - Hydrolysate Silver and Zinc Concentrations for pH 7 Hydrolysis

					SILVER				ZINC		
SAMPLE	TEST	ANALYSIS DATE	DILUTION	SOL'N CONC (ppb)	AMNT RELSED (ug/mg)	AVERAGE (ug/mg)	RPD/RSD	SOL'N CONC (ppb)	AMNT RELSED (ug/mg)	AVERAGE (ug/mg)	RPD/RSD
HL-0A-7	0	3/29/91		189	0.21	0.20	10	387	0.42	0.39	15
HL-0B-7	0	3/29/91		197	0.19			379	0.36		
HL-2A-7	2	3/29/91		207	0.20	0.21	5	342	0.33	0.35	11
HL-2B-7	2	3/29/91		205	0.21	0.21		355	0.37	0.00	
HL-4A-7	4	3/29/91		199	0.22			305	0.34		
HL-4B-7	4	3/29/91		229	0.16	0.19	32	335	0.23	0.29	40
HL-8A-7	8	4/9/91		187	0.17	0.17		248	0.23		
HL-8B-7	8	4/9/91		190	0.17	0.17	0	257	0.23	0.23	0
HL-16A-7	16	4/15/91		172	0.14	014		163	0.14	0.14	
HL-16B-7	16	4/15/91		185	0.14	0.14	0	170	0.13	0.14	7
HL-24A-7	24	4/29/91		187	0.21	0.00	40	196	0.22	0.01	40
HL-24B-7	24	4/29/91		196	0.19	0.20	10	196	0.19	0.21	10
HL-30A-7	30	4/29/91		192	0.23	2.24		315	0.38		
HL-30B-7	30	4/29/91		194	0.19	0.21	19	453	0.43	0.41	12

<sup>\* -</sup> Outlier not used in calculating average

Table 5 - Sample Weights and pH Values for pH 9 Hydrolysis Samples

SAMPLE	POWDER WT (mg)*	INITIAL pH	TEST	TEST DATE	FINAL pH
HL-0A-9	41.64	9.0	0	3/25/91	9.0
HL-0B-9	38.40	9.0	0	3/25/91	9.0
HL-2A-9	40.17	9.0	2	3/27/91	9.0
HL-2B-9	35.21	9.0	2	3/27/91	9.0
HL-4A-9	42.90	9.0	4	3/29/91	9.0
HL-4B-9	27.31	9.0	4	3/29/91	9.0
HL-8A-9	35.19	9.0	8	4/2/91	9.0
HL-8B-9	42.04	9.0	8	4/2/91	9.0
HL-16A-9	41.74	9.0	16	4/10/91	9.0
HL-16B-9	37.92	9.0	16	4/10/91	9.0
HL-24A-9	30.41	9.0	24	4/18/91	9.0
HL-24B-9	37.25	9.0	24	4/18/91	9.0
HL-30A-9	39.25	9.0	30	4/24/91	9.0
HL-30B-9	27.42	9.0	30	4/24/91	9.0

<sup>\* -</sup> Anhydrous basis (corrected for 16.82% moisture)

Table 6 - Hydrolysate Silver and Zinc Concentrations for pH 9 Hydrolysis

				SILVER				ZINC		
SAMPLE	TEST DAY	ANALYSIS DATE DILUTION	SOL'N CONC (ppb)	AMNT RELSED (ug/mg)	AVERAGE (ug/mg)	RPD/RSD	SOL'N CONC (ppb)	AMNT RELSED (ug/mg)	AVERAGE (ug/mg)	RPD/RSD
HL-0A-9	0	3/29/91	30	0.022	0.024	17	8	0.0058	0.0079	53
HL-0B-9	0	3/29/91	33	0.026	0.024		13	0.010	0.0075	33
HL-2A-9	2	3/29/91	46	0.034			71	0.053		
HL-2B-9	2	3/29/91	39	0.033	0.034	3	72	0.061	0.057	14
HL-4A-9	4	3/29/91	49	0.034			117	0.082		
HL-4B-9	4	3/29/91	49	0.054	0.044	45	142	0.16	0.12	65
HL-8A-9	8	4/9/91	115	0.098			457	0.39		
HL-8B-9	8	4/9/91	95	0.068	0.083	36	343	0.24	0.32	47
HL-16A-9	16	4/15/91	184	0.13			888	0.64		
HL-16A-9	16	4/15/91	171	0.12	0.13	8	852	0.61	0.63	5
HL-16B-9 HL-16B-9	16 16	4/15/91 4/15/91	42 34	0.033* 0.027*			84 82	0.066* 0.065*		
HL-24A-9	24	4/29/91	117	0.12	044		640	0.63	0.50	
HL-24B-9	24	4/29/91	118	0.095	0.11	23	516	0.42	0.53	40
HL-30A-9	30	4/29/91	164	0.13			956	0.73		
HL-30B-9	30	4/29/91	202	0.22	0.18	26	1076	1.18	1.03	26
HL-30B-9	30	4/29/91 1:10	184	0.20			1090	1.19		

<sup>\* -</sup> Outlier not used in calculating average

Table 7 - Results for Hydrolysis Study Negative Controls (Blanks)

SAMPLE	INITIAL pH	TEST DAY	TEST DATE	FINAL pH	ANALYSIS DATE	SILVER (ppb)	ZINC (ppb)
BL-0-5	5.0	0	3/25/91	5.0	3/29/91	ND	4
BL-2-5	5.0	2	3/27/91	5.0	3/29/91	29	10
BL-16-5	5.0	16	4/10/91	5.0	4/15/91	ND	101
BL-30-5	5.0	30	4/24/91	5.0	4/29/91	ND	25
BL-0-7	6.9	0	3/25/91	6.9	3/29/91	ND	19
BL-4-7	6.9	4	3/29/91	6.9	3/29/91	ND	13
BL-24-7	6.9	24	4/18/91	6.9	4/29/91	ND	15
BL-30-7	6.9	30	4/24/91	6.9	4/29/91	ND	16
BL-0-9	9.0	0	3/25/91	9.0	3/29/91	ND	2
BL-8-9	9.0	8	4/2/91	9.0	4/9/91	8	11
BL-30-9	9.0	30	4/24/91	9.0	4/29/91	ND	8

Table 8 - Results for Hydrolysis Study Positive Controls (Spikes)

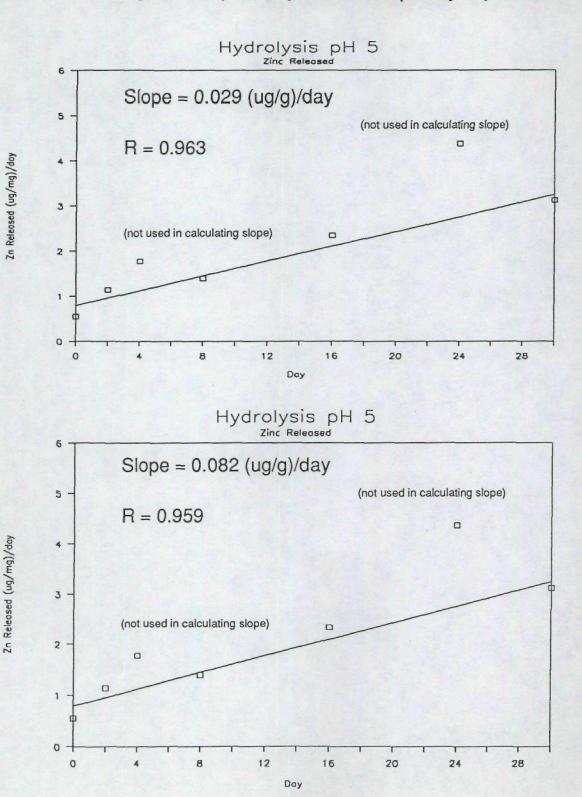
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	INITIAL	TEST	TEST	FINAL	ANALYSIS		SILVER	ZINC	SILVER	ZINC
SAMPLE	pH	DAY	DATE	pН	DATE	FACTOR	(ppb)	(ppb)**	%	%
SP-0-5	5.0	0	3/25/91	5.0	3/29/91		14	131	28	131
SP-0-5-Ag	5.0	0	4/1/91	5.0	4/9/91		58	33*	116	
SP-8-5	5.0	8	4/2/91	5.0	4/9/91		18	114	36	114
SP-8-5-Ag	5.0	8	4/9/91	5.0	4/9/91		50	133*	100	
SP-30-5	5.0	30	4/24/91	4.5	4/29/91		7	100	14	100
SP-30-5-Ag	5.0	30	5/1/91	5.0	5/15/91		35	27*	70	
SP-0-7	6.9	0	3/25/91	6.9	3/29/91		17	126	34	126
SP-0-7-Ag	6.9	0	4/1/91	6.9	4/9/91		57	34*	114	
SP-2-7	6.9	2	3/27/91	6.9	3/29/91		15	106	30	106
SP-2-7-Ag	6.9	2	4/3/91	6.9	4/9/91		45	112*	90	
SP-4-7***	6.9	4	3/29/91	6.9	3/29/91		33	133	66	133
SP-16-7	6.9	16	4/10/91	6.9	4/15/91		ND	184	0	184
SP-16-7-Ag	6.9	16	4/17/91	6.9	4/29/91		16	29*	32	
SP-30-7	6.9	30	4/24/91	6.9	4/29/91		10	121	20	120
SP-30-7-Ag	6.9	30	5/1/91	6.9	5/15/91		12	28*	24	
SP-0-9	9.0	0	3/25/91	9.0	3/29/91		9	120	18	120
SP-0-9-Ag	9.0	0	4/1/91	9.0	4/9/91		61	27*	122	120
SP-4-9	9.0	4	3/29/91	9.0	3/29/91		27	136	54	136
SP-4-9-Ag	9.0	4	4/5/91	9.0	4/9/91		39	107*	78	
SP-24-9	9.0	24	4/18/91	9.0	4/29/91		2	150	4	150
SP-24-9-Ag	9.0	24	4/25/91	9.0	4/29/91		15	29*	30	
SP-30-9	9.0	30	4/24/91	9.0	4/29/91		6	133	12	133
SP-30-9-Ag	9.0	30	5/1/91	9.0	5/15/91		9	24*	18	

<sup>\* -</sup> Zinc background interference. Not corrected for blank concentration

<sup>\*\* -</sup> Corrected for blank concentration on corresponding day

<sup>\*\*\* -</sup> Contigency sample SP-C-7 used for analysis

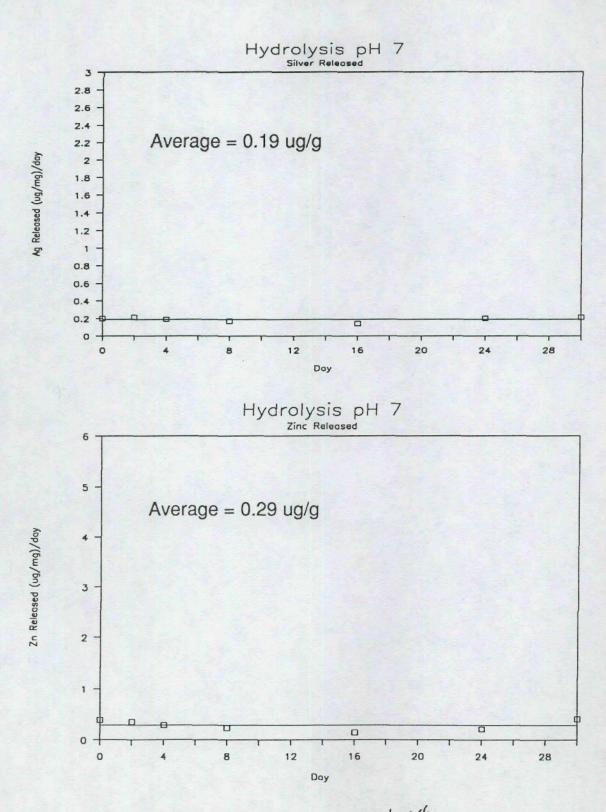
Figure 1 - Graphical Representation of pH 5 Hydrolysis Results



**Arthur D Little** 

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Figure 2 - Graphical Representation of pH 7 Hydrolysis Results



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Figure 3 - Graphical Representation of pH 9 Hydrolysis Results

